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Reverse osmosis in food processing

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Agricultural Research Service
UNITED STATES DEPARTMENT OF AGRICULTURE

THE SYMPOSIUM ON REVERSE OSMOSIS IN FOOD PROCESSING was cosponsored by the National Canners Association and the U.S. Department of Agriculture. It was attended by representatives of the food industry and by suppliers of reverse osmosis equipment.

The sumposium was organized in the Engineering and Development Laboratory of the Western Utilization Research and Development Division of Agricultural Research Service by J. P. Clark, who also compiled the proceedings.

Fred Senti, Deputy Administrator of Agricultural Research Service for Nutrition, Consumer and Industrial Use Research, opened the conference with welcoming remarks directed to the role the Division had played in developing reverse osmosis.

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August 1969

CONTENTS

REVERSE OSMOSIS IN FOOD PROCESSING

AN OVERVIEW

R. L. Merson, Chemical Engineer[1/]
Western Utilization Research and Development Division
Agricultural Research Service, USDA, Albany, Calif.

Membrane separations are being considered seriously for use in food processing (1-8). In particular reverse osmosis is well into the research stage in food processing applications. The purpose of this conference was to bring together those familiar with food problems and those acquainted with reverse osmosis in hopes of exchanging information between the two groups. To achieve this goal we have tried to limit our discussions to applications of reverse osmosis and to the problems specifically related to foods rather than to the many theoretical aspects of transport through membranes.

Reverse osmosis.--Reverse osmosis is a membrane process which uses hydrostatic pressure to separate components of a solution (fig. 1). For example, because of the dissolved solutes such

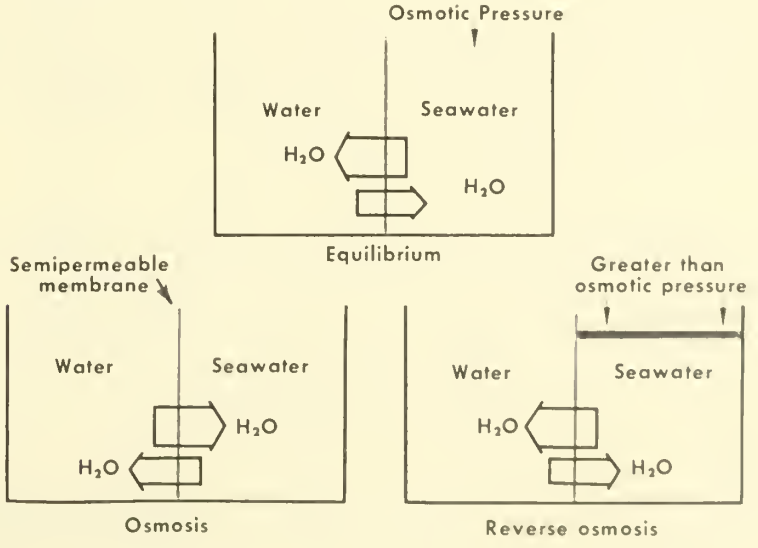

Figure 1.--Osmosis and reverse osmosis.

1/ Present address: Department of Food Science, University of California, Davis.

as sugars and acids, a fruit juice has a property called the osmotic pressure. Using a semipermeable membrane which will pass water but will not allow the osmotic solutes to pass, we can concentrate the juice by applying hydrostatic pressure in excess of the osmotic pressure. The two basic problems in reverse osmosis are how fast can you get the water out and how well can you keep the solutes in?

Applications.--The remaining formal papers in these proceedings are examples of food applications of reverse osmosis. The applications can be divided into three categories each of which makes use of one or more of the following advantages: (1) Simple concept, (2) no phase change--lower energy costs than heat transfer methods, (3) ambient temperatures, and (4) operation remote from steam generation plant. Concentration uses a tight membrane to remove only water, leaving solutes and aroma behind with no thermal damage. Purification (or fractionation) uses a special membrane to take out some unwanted solute with the water. Reverse osmosis can be used on waste disposal problems to recover water cheaply from dilute waste streams or to concentrate the waste material sufficiently to make use of conventional disposal methods feasible.

Membranes.--The semipermeable membranes are thin, fragile, plastic films. The most successful material so far is cellulose acetate. These membranes are made--by the process developed at the University of California, Los Angeles, by Loeb and Sourirajan (9) and Manjikian (10)--to have a very thin, dense skin about 0.2 μ thick. This skin provides the semipermeability of the membrane and also the resistance to water transport. Only because this skin is so thin are acceptable permeation rates achieved. The rest of the membrane is about 100 μ thick and is very open and porous. It provides support for the skin.

These cellulose acetate membranes have some limitations. The porous substructure tends to collapse under very high pressure. The cellulose acetate can be hydrolyzed by extremes of pH. Some concentrated organic solvents such as alcohol render the separating qualities of the membranes ineffective, and furthermore, cellulose acetate simply cannot perform every separation which we would like. Research to overcome these difficulties has led to promising results for making Loeb-type membranes of other materials (Amicon) and for making artificial "skins" of ultrathin semipermeable membranes which are then supported on a second very porous nonselective membrane (North Star). Glass membranes (McDonnell-Douglas) are also promising. In spite of its deficiencies, cellulose acetate is quite versatile and will not soon be replaced. We hope to discuss some of these problems in the section on membranes.

Two factors describe membrane performance: permeation rate and separation or solute retention. For cellulose acetate membranes, these two factors are interdependent. A "tight" membrane retains aroma and sugar well, but has a slow water-removal rate. To increase the water-removal rate usually involves a sacrifice in solute retention. To achieve control over these variables we simply heat the cellulose acetate membrane in hot water for a few minutes before using it.

The maximum permeation rate that can be achieved at 1,000 p.s.i., that is, the permeation with pure water on both sides of the membrane (intrinsic pure water rate), is plotted in figure 2

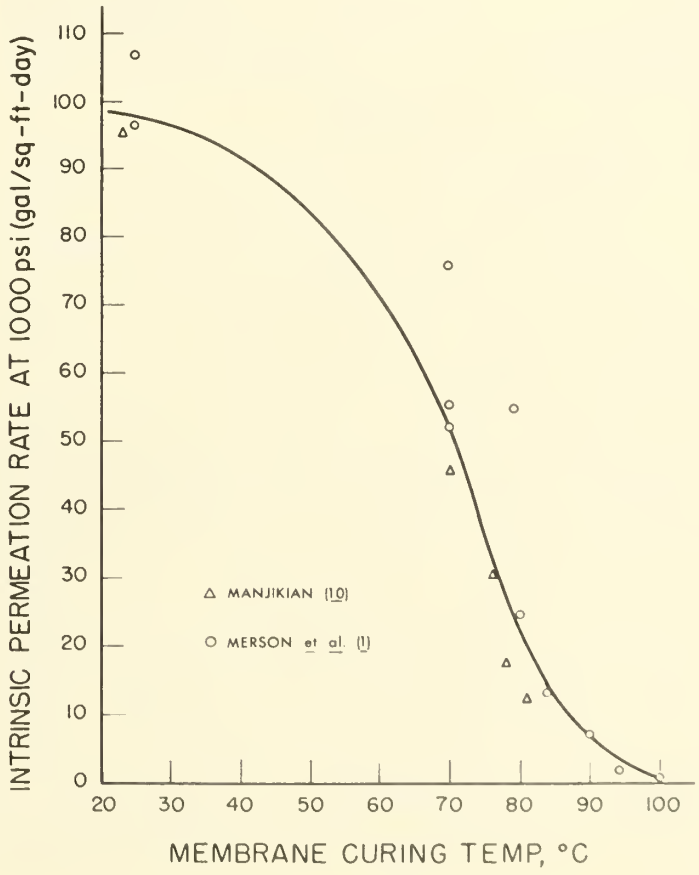

Figure 2.--Intrinsic permeation rate of Loeb type cellulose acetate membranes. The flux can be controlled by curing the membranes in hot water.

as a function of the curing temperature of the membrane. The higher the curing temperature, the tighter the membrane, and the lower the rate at which it will let water pass.

The ordinate in figure 2 is used as the abscissa in figure 3 where the ratio of solids concentration in the permeating water to

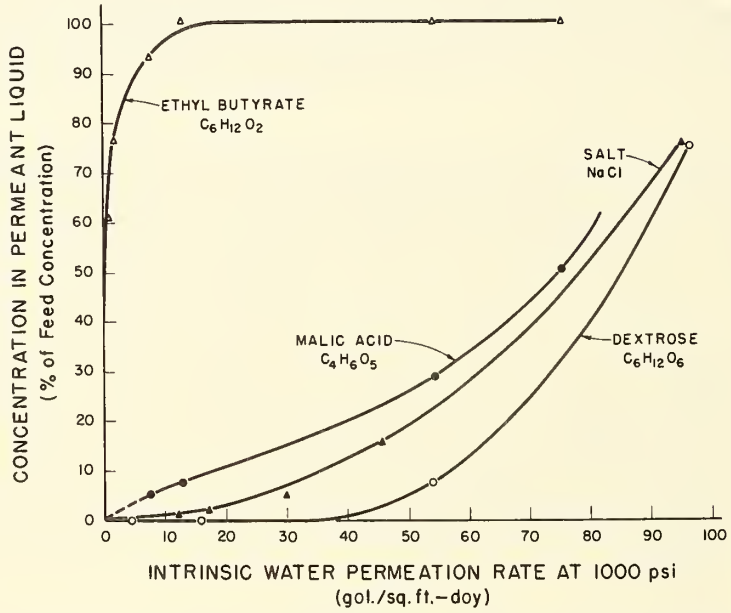

Figure 3.--Solute retention with cellulose acetate membranes. Data of Manjikian (10) (salt) and Merson and others (1) (dextrose, malic acid).

solids concentration in the feed liquid is plotted against the intrinsic pure water permeability of each membrane. Notice that for up to about 30 to 40 gal. per sq. ft. per day, no dextrose (or sucrose) appears in the permeant, although considerable salt does. The ester is representative of the aroma of fruit juices. It is important to notice that the concentration ratio for the ester never is higher than 1, even for the most open membranes. Thus, if you prepare a fourfold concentrate by removing 75 percent of the water from a fruit juice, at most you remove only 75 percent of the aroma. The remaining 25 percent is sufficient to give the reconstituted concentrate a good aroma, accounting in part for the high quality of the concentrate. Actually, aroma retention in fruit juices is somewhat better than is indicated here for a synthetic mixture.

Figure 4 is a chromatographic representation of the aroma over fresh orange juice and over the water that permeates the membrane. This illustration shows that the oil soluble aroma molecules in orange juice--mostly hydrocarbons here--do not permeate the membrane. But a fraction of the water soluble compounds--small esters, aldehydes, and alcohols--do pass through.

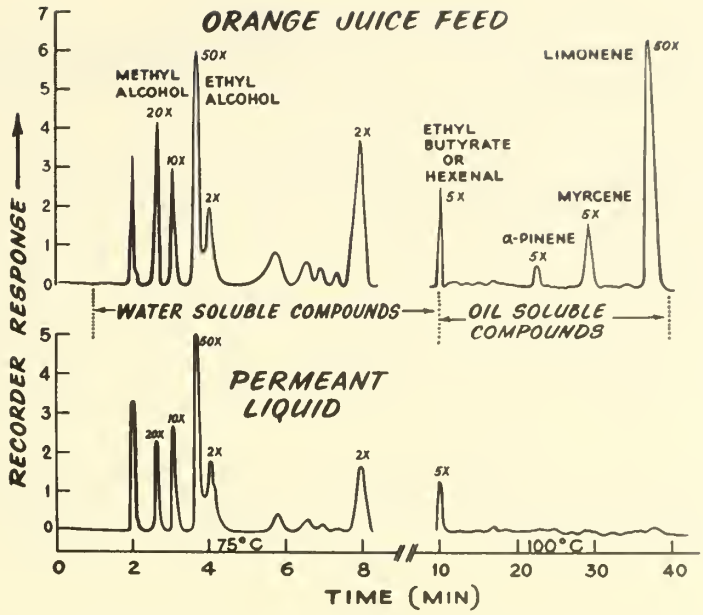

Figure 4.--Chromatograph of volatile aroma of orange juice.

Physical requirements for a reverse osmosis system.--For a perfectly stirred system, the permeation rate is given by

$$\text{RATE} = KA(\Delta p - \Delta\pi).$$

K is the reciprocal of the membrane resistance and is equivalent to a permeation rate coefficient; A is the membrane area; Δp is the applied hydrostatic pressure; and for a semipermeable membrane $\Delta\pi$ is approximately the osmotic pressure of the feed solution. In general, K is a very small number so that large membrane areas are necessary.

The quantity $(\Delta p - \Delta\pi)$ is the driving force for water removal. Osmotic pressures (table 1) range from a few pounds per square inch for dilute feeds of high molecular weight substances

TABLE 1.--Osmotic pressures

	Concentration	Osmotic pressure
		P.s.i.
Processors effluent	0.5 pct.	250
Sea water	3.5 pct.	350
Sugar beet thin juice	20° Brix	500
Tomato paste	33° Brix	1,000
Citrus juice	10° Brix	215
Citrus juice concentrate	45° Brix	1,500

up to 1,500 p.s.i. for fruit juice concentrates. At least in some cases fairly high hydrostatic pressures are necessary to overcome the osmotic pressure. The membrane must be properly supported to withstand these pressures.

Concentration polarization.--As the water is removed, the sugar molecules remain behind raising the osmotic pressure of the feed. If the feed is not perfectly mixed, sugar will accumulate near the membrane and water will have to diffuse through the high-sugar layer as well as through the membrane. This phenomenon is called concentration polarization. It results in a permeation rate which is considerably lower than the maximum permitted by the membrane alone. So it is very important to have good mixing.

One way to provide mixing is to place another membrane parallel to the first and cause the feed to flow through the channel thus created (fig. 5). The permeation rate or flux is

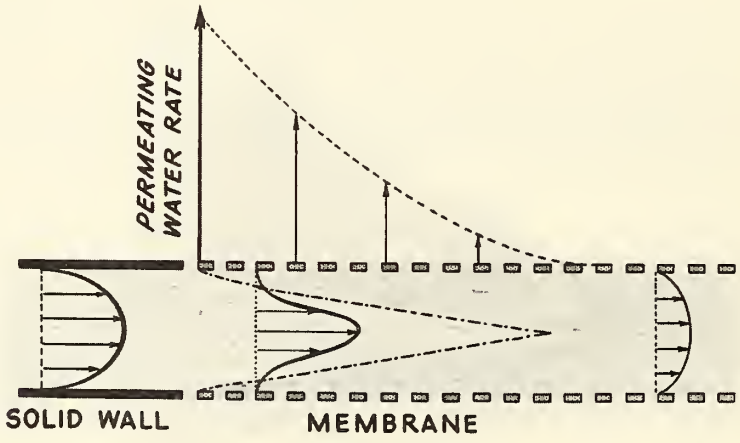

Figure 5.--Model channel.

schematically represented above the semipermeable part of the channel. At first the flux is limited only by the membrane and is quite high. But farther downstream the flux diminishes as concentration polarization adds additional resistance to water transport.

Concentration polarization is aggravated with foods because the viscosity of the solution increases with solids concentration. The figure illustrates what happens to the flow. The flow here is laminar and enters the channel with the characteristic parabolic velocity profile. As sugar accumulates near the membrane, the viscosity rises there and downstream the velocity profile becomes distorted. In fact, the flow stagnates over a considerable fraction of the channel thickness with the dilute feed flowing

rapidly along the center line of the channel. Water must diffuse through this stagnant boundary layer to reach the membrane.

TABLE 2.--Apparent viscosity of some liquid foods[1/]

Substance	Single strength		Concentrated	
	Concentration	Viscosity	Concentration	Viscosity
	°Brix	C.p.	°Brix	C.p.
Sucrose solution	10	1.1	44	6.6
Grape juice	18	2	44	20
Tomato juice	5	3	33	2,000
Egg white	12	6	30	40

Note: Viscosity of water 0.9 c.p.
Viscosity of SAE 30 oil (60°F.) 400 c.p.

1/ At 25°C., shear rate of 500 $sec.^{-1}$.

Table 2 shows the magnitude of the viscosity for some liquid foods both at single strength concentration as at the entrance or near the center line of the channel, and at an elevated concentration as at the membrane surface or near the channel outlet. It is important to note two points: (1) the disparity in viscosity between the membrane surface and the center line, which causes stagnation near the membrane, and (2) the exponential increase in viscosity with concentration. When the permeation rate is limited by polarization, the wall concentration is set by the system pressure (that concentration which gives rise to an osmotic pressure equal to the system pressure). Thus if egg white were concentrated at 500 p.s.i. the wall concentration might be near 50 percent protein with an apparent viscosity of 1,200 c.p.

To summarize the physical requirements of a reverse osmosis system (fig. 6), you need a pressurizing pump for the feed, you need large membrane area in equipment which will withstand high pressures and properly support the membrane, and you need to provide good mixing. In addition you must make provision for taking the equipment apart for cleaning. Besides being viscous, foods can clog small channels. Suspended materials will settle out on the membrane, and you must prevent microbial growth.

Market potential.--The annual volume and wholesale market value of the five commodities listed in table 3 should be considered rough estimates. The tomato value is for fruit cost only and should probably be around 200 million dollars on the same basis of wholesale market value as the other products. The tomato juice figure includes catsup, purees, pastes, and unconcentrated canned juice. The orange juice figures are for juice processed to commercial fourfold concentrate only. Egg white is not presently

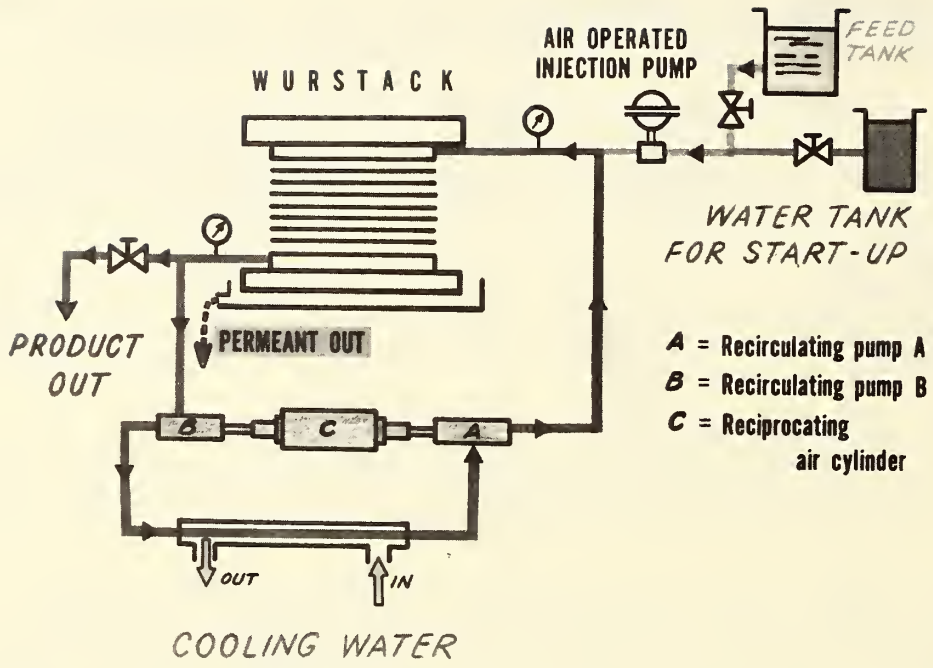

Figure 6.--Typical reverse osmosis system for food processing.

TABLE 3.--Potential market

Food	Annual volume	Wholesale market value
	Billion pounds	Million dollars
Tomato juice[1/]	5.0	90[2/]
Orange juice[3/]	3.2	300
Egg white[4/]	0.2	30
Whey[5/]	22.0	--
Coffee	--	--

1/ National Canners Association (11). 2/ Fruit cost only. 3/ National Association of Frozen Food Packers (12); Florida Citrus Commission (13). 4/ Lowe and others (8). 5/ Marshall and others (6).

concentrated at all. Whey is rich in protein and food grade lactose but in unprocessed condition is a waste disposal problem. Over 25 percent of coffee in the United States is sold as dry powder.

Figures in table 4 indicate that about 8 to 10 percent of the retail cost of a product is attributable to direct processing costs (exclusive of warehousing, sales, and packaging costs).

TABLE 4.--Orange juice costs[1/]

Costs	Cents	Dollars
Grower's costs per doz. cans (6 oz.):		
Labor, power, equipment	16.80	
Taxes	2.40	
Interest	13.20	
Fertilizer, spray	13.20	
Miscellaneous	3.60	
Picking costs	9.60	
Grower's margin	13.68	
	72.48	0.7248
Processor's costs:		
Power, light, water, maintenance, royalties, depreciation, taxes, rent	9.24	
Labor	5.28	
Warehousing	3.00	
Sales, administrative costs[2/]	18.00	
Cost of cans	24.00	
	59.52	0.5952
Transportation costs		0.1488
Retailing costs including profit		0.3912
Total		1.86

1/ From Frozen Food Executive Bulletin, August 25, 1967 (14). 2/ Includes cost of brokers and wholesalers.

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CONCENTRATING EGG WHITE

Edison Lowe, Head, Equipment Investigations
Western Utilization Research and Development Division
Agricultural Research Service, USDA, Albany, Calif.

Egg white concentration is perhaps the most interesting food application of reverse osmosis simply because there aren't many other ways this material can be concentrated without denaturing the proteins in the albumen. Egg white is used in the candy and baking industry primarily because of its ability to form foams stable enough to support relatively large quantities of flour or sugar or both. This most important functional property is impaired by high processing temperatures or by physical treatments involving shear stresses. Reverse osmosis does not involve elevated processing temperatures but does under ordinary circumstances subject the product to intense shear when it is suddenly released from the high pressure zone. To avoid mechanical stresses of this type, a special bulk transfer device was developed to insure gradual withdrawal of the concentrate from the reverse osmosis apparatus.

Let's take a look at this rather important modification of a conventional reverse osmosis (fig. 1). The important part of

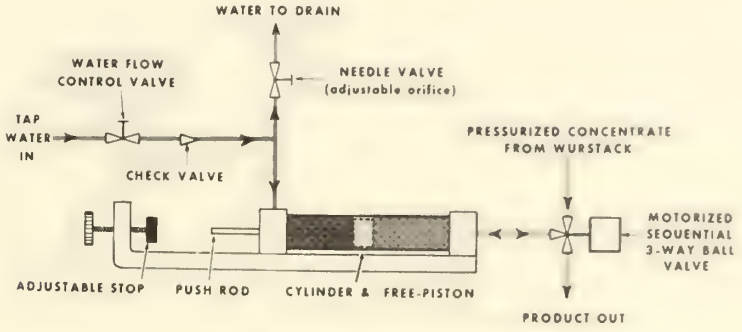

Figure 1.--Zero shear discharge device.

the bulk transfer device involves a cylinder and a free-piston. The piston is activated at one end by the concentrate discharging under pressure from the reverse osmosis apparatus, and at the opposite end by tapwater under house pressure. A sequential rotary ball valve periodically admits concentrate to one end of the cylinder, pushing the piston against the water trapped in the opposite end of the cylinder. An adjustable orifice controls the rate at which water is drained out of the cylinder and, therefore, the rate at which the concentrate can enter the cylinder at the opposite end. A check valve prevents water from backing up into the house water supply line.

When the cylinder is filled with concentrate, the three-way rotary valve is advanced to the second position, connecting the cylinder to a product outlet. This depressurizes the concentrate so that it can be gradually pushed out of the cylinder by the water on the other side of the piston. A valve in the house water supply line controls the rate at which the concentrate will flow out at the opposite end. The cycle is repeated when the rotary valve is returned to its first position.

A push rod connected to the piston and extending out through the water end of the cylinder travels against an adjustable stop to permit control of the product discharge volume. The rod also actuates a microswitch, not shown in this diagram, that energizes the sequential rotary valve to depressurize the concentrate trapped in the cylinder.

The degree of concentration is automatically controlled by maintaining a constant ratio of permeant volume to concentrate volume. An automatic syphon is used to trap a fixed volume of permeant. When the syphon is full, the permeant closes the

contacts of a zero current relay which in turn energizes the rotary valve to start the discharge sequence that we've just described.

Functionally, proteins are the essential ingredient in egg white and because of their size are easily retained, even by the most open membrane. The cellulose acetate membranes that were used to concentrate egg white were left untempered for maximum flux. Initial pure water permeation rate, a measure of the tightness of the membrane, was 62 gal./sq. ft.-day at 400 p.s.i.

The initial pH of raw liquid egg white is approximately 9.0. To study the effect of pH on the functional properties of the concentrate, a portion of the feed material was adjusted to pH 7.0 by the addition of a 10-percent lactic acid solution.

I've been intimating all along here that we can produce an egg white concentrate with good functional properties, so I guess we'd better come up with some data that tend to confirm this.

Figure 2 shows the whipping characteristics of meringues made from the control and from the reconstituted concentrates.

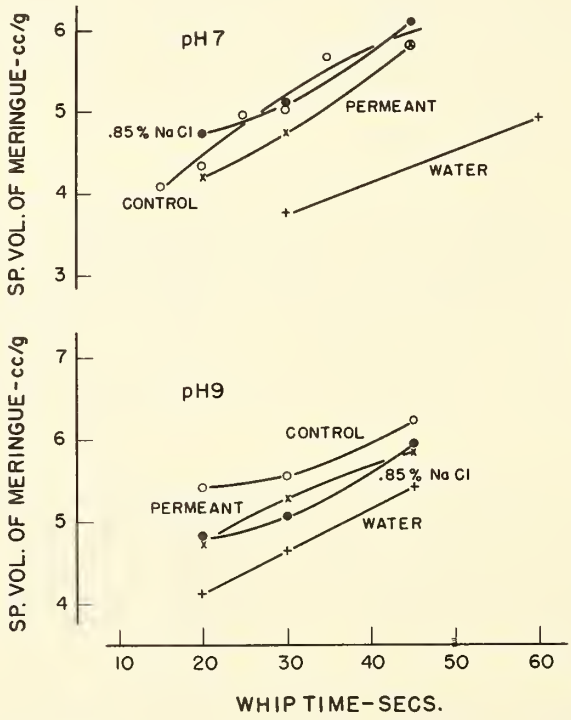

Figure 2.--Effect of permeant, saline solution and water on meringue volume.

The concentrated samples were reconstituted in three different ways: (1) with water, (2) with an 0.85-percent NaCl solution, and (3) with their own permeants.

Other investigators have noted the effect of salt level on the whipping property of reconstituted egg white concentrate. The ionic strength of a concentrate produced by membrane separation is lowered by removal of salts. The ionic strength is restored by use of a saline solution to reconstitute the concentrated albumen. Commercially, the same results can be achieved by adding salt to the concentrated product.

Reconstituting with permeant returns to the sample all of the solutes that passed through the membrane along with the water.

In this figure, meringue volume is shown as a function of whip time. You can see that there is substantially no difference in the performance of the control and the pH 7 concentrate samples reconstituted either with an 0.85-percent saline solution or its own permeant. On the other hand, the sample reconstituted with water took twice as long to reach the same specific volume.

Samples of the pH 9 concentrate reconstituted with the 0.85-percent saline solution and with their own permeants required about 20 to 40 percent more whipping time than did the control. The water-reconstituted sample took twice as long to whip to the same specific volume as the control.

Figure 3 shows the results of a large angel cake baking test. From a commercial standpoint it would not be practical to

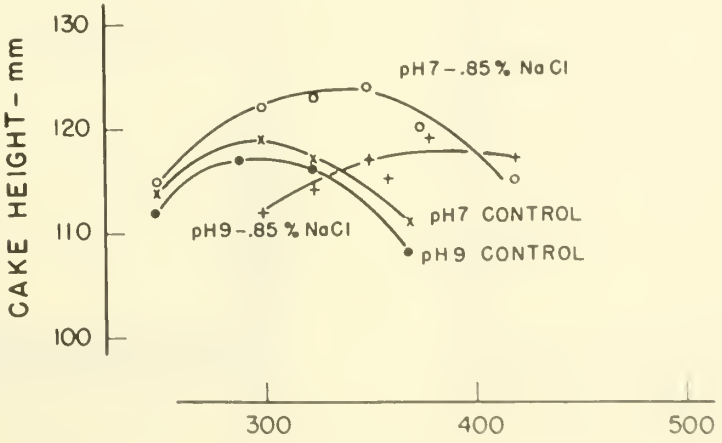

Figure 3.--Effect of saline solution on angel cake height.

reconstitute the concentrate with permeant, nor would it be desirable to reconstitute with water because the resulting albumen would not make a good meringue. The data shown are, therefore, for concentrate samples reconstituted with an 0.85-percent saline solution.

Here, cake height is shown as a function of whip time. For the same whip time, then, the pH 7 concentrate yielded cakes of greater volume than those from the control. Maximum cake volume was 4.2 percent greater for the concentrate sample than for the control. Cake volume for the concentrate was greater at all whip times but the optimum whip time was somewhat longer (350 vs. 300 sec.).

Performance of the reconstituted pH 9 concentrate was not as good as for the control. Cake volume was smaller at a whip time of 280 sec., which was optimum for the control. Volume is equal at optimum whip times. Texture was good for all of the cakes that were not underwhipped or overwhipped. Figure 4 shows sample slices taken from cakes with approximately optimum whip.

The selectivity of the high flux membranes used to concentrate the egg white is shown in tables 1 and 2. Table 1 shows

TABLE 1.--Gross chemical composition of liquid egg white

	Pct.
Water	87.7
Solids	12.3
Sodium chloride	0.3
Protein	10.7
Free glucose	0.38

TABLE 2.--Selectivity of membrane

Constituents	Control	Concentrate	Permeant
Total solids, pct.	11.9	28.9	1.07
Glucose, pct.	0.40	0.50	0.28
Sodium, pct.	0.18	0.23	0.19
Potassium, pct.	0.17	0.20	0.15
Phosphorus, mg./ml.	0.12	0.30	0.001
Calcium, mg./ml.	0.061	0.097	0.023
Amino nitrogen, mg./ml.	--	--	nil
Total nitrogen, mg./ml.	16.76	48.9	0.03

the gross chemical composition of raw liquid egg white. You can see that albumen contains about 12 percent solids, and of the solids about 0.3 percent is NaCl, 10.7 percent is protein, and about 0.4 percent is free glucose. Table 2 shows the chemical

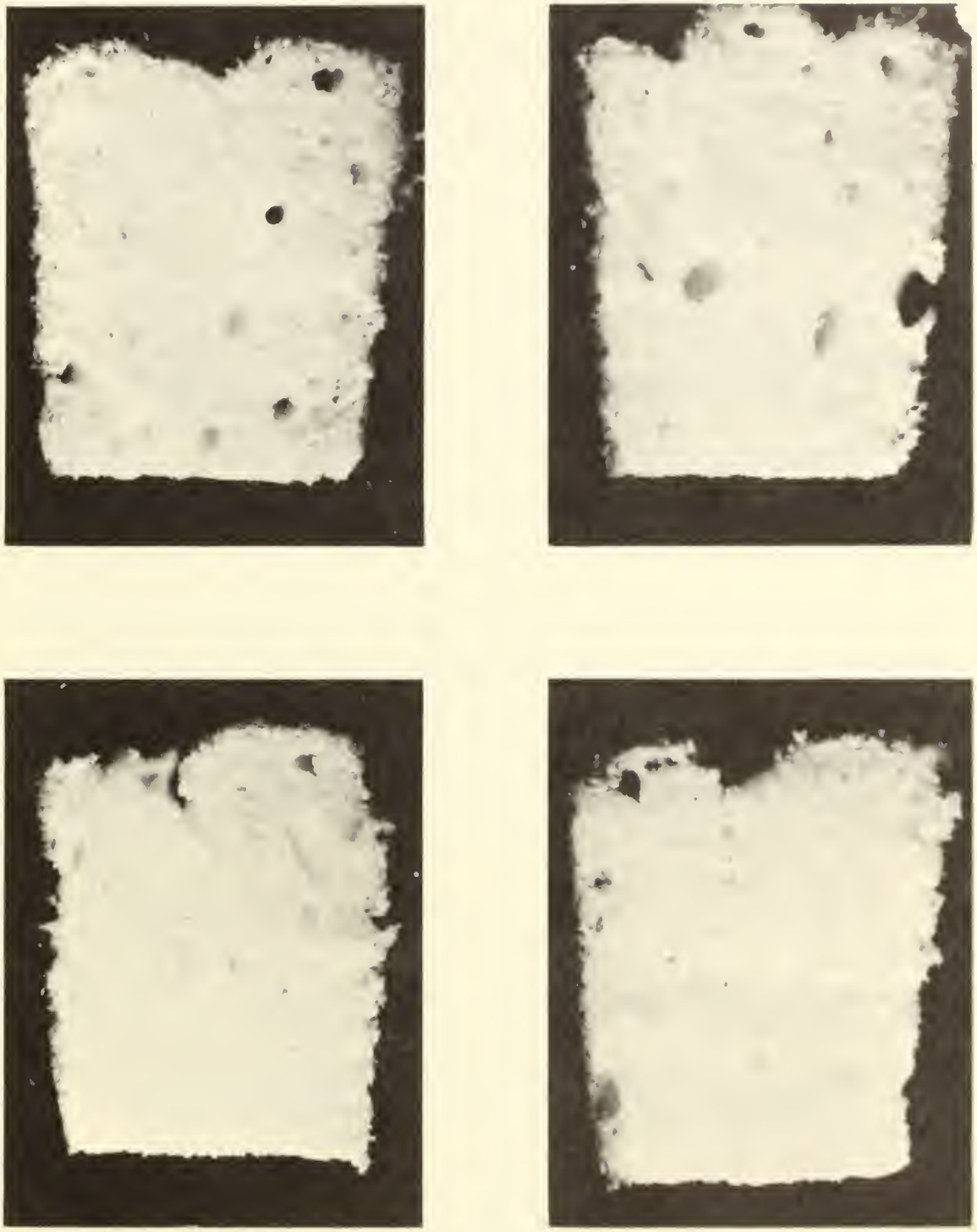

Figure 4.--Slices from test cakes. Top left: pH 7 control. Top right: pH 7 + NaCl. Bottom left: pH 9 control. Bottom right: pH 9 + NaCl.

analyses of the feed material, the concentrate, and the permeant. There was little difference in results between the natural feed and the neutralized feed. The membranes retain nearly 100 percent of the large protein molecules. Under the conditions of the experiments about 40 to 50 percent of the glucose, about 50 percent of the sodium and potassium salts, and about 20 percent of the clacium were removed from the feed material. This is consistent with the observation of others that divalent salts are better retained than monovalent salts.

The fact that about 40 to 50 percent of the free glucose is removed with the water is significant because it reduces the desugaring requirements for a solids product. In the production of egg white powder, the glucose is removed prior to drying to prevent the Maillard reaction and thus improve the storage stability of the dried product. This is accomplished either by controlled fermentation of the glucose or by an enzymatic treatment in which glucose is oxidized to gluconic acid. The fermentation process requires adjustment of the pH from an initial value of 9.0 to 7.0 for optimum growth of the microorganisms. Reducing the glucose content by 40 to 50 percent should certainly lighten the desugaring load.

So far as is known, egg white is not concentrated commercially at the present time. The advantages of a concentrate would be several. For the frozen product, there would be economic advantages in reducing the packaging, freezing, storage, and transportation requirements. For egg white solids, there are potential savings in the cost of removing water by concentration rather than the more expensive drying, and as we pointed out earlier, there are also potential savings in the cost of desugaring because of the reduced vessel and heating requirements.

CONCENTRATION OF MAPLE SAP

J. C. Underwood, Chemist, Maple Investigations
Eastern Utilization Research and Development Division
Agricultural Research Service, USDA, Wyndmoor, Pa.

The manufacture of maple sirup requires that maple sap be concentrated 30 to 40 times depending upon the amount of sugar in it. This concentration is normally done by atmospheric boiling in open pan evaporators (1). The thermal energy required for processing (from wood or fuel oil) costs about 50¢ per gal. of

sirup. Consequently, people in the industry are continually on the alert for cheaper means of doing this evaporation.

At the National Institute of Food Technologists Meeting, Portland, Oreg., in 1966, I reported that we at Eastern Utilization Research and Development Division had successfully applied reverse osmosis on a laboratory scale to the concentration of maple sap in making maple sirup (2). Flavor precursors and sugars were rejected by the membrane. Owing to the need of heat to develop maple flavor and color, maple sap can be concentrated only partially by reverse osmosis. We found that doing 75 percent of the concentration by reverse osmosis and 25 percent by conventional atmospheric boiling was feasible.

Based on this work and on consultation with the manufacturers of membranes, a plant-size reverse osmosis unit was built last year. Designed to handle 10,000 gal. of maple sap a day, by removing 75 percent of the water from the feed, the unit contains eight 10 ft. x 4-in. diameter pressure tubes holding a total of 800 sq. ft. of cellulose acetate membrane in the spirally wound module form. To achieve a constant flow and pressure, a positive displacement screw-type pump is driven by a single phase 7-1/2 h.p. motor. Pressure applied to the modules is controlled by means of an adjustable back pressure regulator located in the concentrate output of the pressure vessels. A special feature of the unit is an in-line ultraviolet sterilizer to control bacterial growth. The unit (EUROC) contains special sampling devices to enable evaluation of the efficiency of the individual pressure tubes. It was tested with a sugar solution prior to installing it in a commercial maple sirup plant for field evaluation. At 600 p.s.i.g. and 60°F., a flux rate of 5.3 gal./day/ft.^{2} was found for the unit (3).

At the sirup plant of J. L. Sipple and Sons, Bainbridge, N.Y., 10,000 gal. of maple sap were handled satisfactorily by EUROC. The reverse osmosis-treated sap produced sirup that could not be differentiated from that conventionally made at the Sipple plant. The analysis of several samples of the "pure" water from maple sap processing is shown in table 1. The sugar lost averaged

TABLE 1.--Analysis of byproduct water
(water removed from maple sap by reverse osmosis)

	Sample No.		
	S-5	S-18	S-29
Specific conductance, micromhos	26	37	37
Total solids, p.p.m.	1,510	380	830
Ash, p.p.m.	19	15	20
Sugar, p.p.m.	79	88	56

1 part in 500 parts of the sugar in the sap. The flux rate at the commercial plant was lower than theoretically estimated. This was partially due to the relatively low temperature (40°-45°F.) of the maple sap feed. Since these tests were run, new modules have become available with flux rates 50 percent higher. At the Sipple plant it was found that 50 percent of the water to be removed in making maple sirup could be feasibly done by reverse osmosis. This is a saving of 54 percent in fuel cost as shown in table 2. With the improved modules it will be feasible to remove 75 percent of the water, thus further lowering the fuel cost.

TABLE 2.--Costs of concentration of maple sap

A.	Removal of 1 gal. of water	
	by reverse osmosis (electricity)	0.06¢
	by thermal distillation (oil fuel)	1.50¢
B.	Concentration of 2.5° Brix sap to 1 gal. of sirup; thermal distillation (oil fuel) combination	49.8¢
	55 percent reverse osmosis	1.1¢
	45 percent distillation	22.4¢
	Total	23.5¢
C.	Savings by partial use of reverse osmosis, 54 pct.	

The field tests showed that technically it is entirely possible to utilize reverse osmosis in making maple sirup. Capital investment cost will determine how soon the maple industry can use the process. Meanwhile we will continue to study membrane life and sanitation procedures with our unit.

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CONCENTRATING AND FRACTIONATING WHEY

W. L. Dunkley, Professor
Department of Food Science and Technology
University of California, Davis

Laboratory and pilot-scale studies of processing whey by reverse osmosis and ultrafiltration are in progress in a number of public and commercial organizations in the United States and in several other countries. In preparing for this discussion, I wrote to a number of these organizations to request brief statements of principal results or appraisals of feasibility of the process, to be included in capsule form here. I have drawn heavily on the replies I received. I wish to thank those who responded so generously to my request for information.

Potential benefits from processing whey by reverse osmosis include economical concentration and selective fractionation. The fractionation can eliminate unwanted salts and adjust proportions of desired constituents, such as lactose and protein. In addition, if the cost of processing is low enough and adaptable to even the smallest cheese factories, the serious waste disposal problem created by cheese factories in many communities will be eliminated.

Typical compositions of cheddar and cottage cheese whey are given in table 1. The low total solids emphasize the need for low-cost concentration to justify recovery of the valuable food solids, mainly protein and lactose. In comparison with cheddar cheese whey, cottage cheese whey is lower in total solids, is higher in ash, and has a higher acidity.

TABLE 1.--Composition of whey[1/]

Components	Cheddar cheese	Cottage cheese
Total solids, pct.	7.31	6.53
Lactose, pct.	5.20	4.39
Protein, pct. N x 6.38	0.87	0.86
Ash, pct.	0.53	0.61
pH	6.0	4.6

1/ From data of McDonough (5).

Table 2 shows the composition of the solids in cheddar cheese whey and in the two fractions obtained by pilot-scale reverse osmosis processing to a concentration of 31.4 percent total solids (i.e., a concentration ratio of 4.3:1). The dry matter in the whey and the concentrate contains about 72 percent lactose and 12 percent protein. Lactose made up 22 percent of the

solids in the permeate, but the concentration of total solids was low--only 0.27 percent. Therefore, less than 1 percent of the total lactose was lost in the permeate. The ash represented almost one-half of the solids in the permeate, and 18.9 percent of the total was eliminated in the permeate.

TABLE 2.--Distribution of solids in fractions from reverse osmosis of cheddar cheese whey[1/]

Components	Composition of dry matter in--			
	Whey	Concentrate	Permeate	Loss in permeate
	Pct.	Pct.	Pct.	Pct.
Lactose	71.2	72.5	22.2	0.8
Protein	11.9	11.6	--	--
Ash	7.3	5.8	48.2	18.9

1/ Calculated from data of McDonough (5). A Havens Type 3A membrane, 700 p.s.i. was used.

Table 3 contains similar data for cottage cheese whey concentrated to 32.6 percent solids by reverse osmosis. The solids in the whey and concentrate were lower in lactose but higher in protein and ash than for cheddar cheese whey. The ash was equal to 60 percent of the dry matter in the permeate. Elimination in the permeate of constituents measured as ash was greater for cottage cheese whey (23.7 percent) than for cheddar cheese whey (18.9 percent).

TABLE 3.--Distribution of solids in fractions from reverse osmosis of cottage cheese whey[1/]

Components	Composition of dry matter in--			
	Whey	Concentrate	Permeate	Loss in permeate
	Pct.	Pct.	Pct.	Pct.
Lactose	67.3	68.0	23.3	1.3
Protein	13.2	12.3	--	--
Ash	9.3	7.9	60.0	23.7

1/ Calculated from data of McDonough (5). A Havens Type 3A membrane, 700 p.s.i. was used.

The data in table 4 show that even the salt constituents can be fractionated by reverse osmosis. The relatively open Loeb-type membrane used in this experiment passed most of the monovalent salts and about half of the lactates and inorganic phosphates but retained most of the calcium and citrate.

Table 5 contains results of an experiment that illustrate the fractionation of salts in a different whey. Concentrating the whey to about double the original total solids eliminated almost half of the sodium chloride but smaller proportions of other salt constituents.

TABLE 4.--Rejection rates for selected constituents during reverse osmosis[1/] of cottage cheese whey

Constituent	Rejection ratio[2/]
Sodium	0.14
Potassium	.10
Calcium	.76
Phosphate	.44
Citrate	.85
Lactate	.44
Lactose	.90

1/ Data of Lowe and Dunkley (3). Loeb-type membrane in Wurstack apparatus, 500 p.s.i., was used.

2/ Rejection ratio = $1 - \frac{\text{Conc. in permeate}}{\text{Conc. in concentrate}}$

TABLE 5.--Losses of salt constituents in the permeate during reverse osmosis of cottage cheese whey[1/]

Constituent	Pct. of total in the permeate
Chloride (as NaCl)	45.9
Calcium	16.3
Ash	20.8
Lactose	7.3

1/ Data of Attebury (1). A Havens Type 2A11 membrane, 500 p.s.i. was used, concentrating whey from 6.5 to 12.6 percent total solids. Average percent total solids in permeate = 0.6 percent.

If very open membranes are used, the proteins and lactose can be separated. Figure 1 illustrates a proposal for processing whey in two stages, the first to concentrate the protein but pass other constituents, and the second to concentrate and fractionate the ultrafiltrate (permeate) from the first stage. In a laboratory evaluation of this process, the protein was concentrated tenfold in the first stage. In the second, the concentration of total solids was increased from 5.6 to 13.9 percent, yielding permeate with 0.12 percent total solids and biological oxygen demand (BOD) of less than 1,000 p.p.m.

The data in table 6 (4) illustrate a different approach for preparing high-protein fractions. In this experiment, the whey was concentrated by reverse osmosis in two stages to 53.3 percent total solids, which resulted in crystallization of part of the lactose. The suspension was centrifuged, and three fractions were separated. The precipitate was mostly (90 percent) lactose. In the other two fractions, the solids contained 47.0 and 56.8 percent protein.

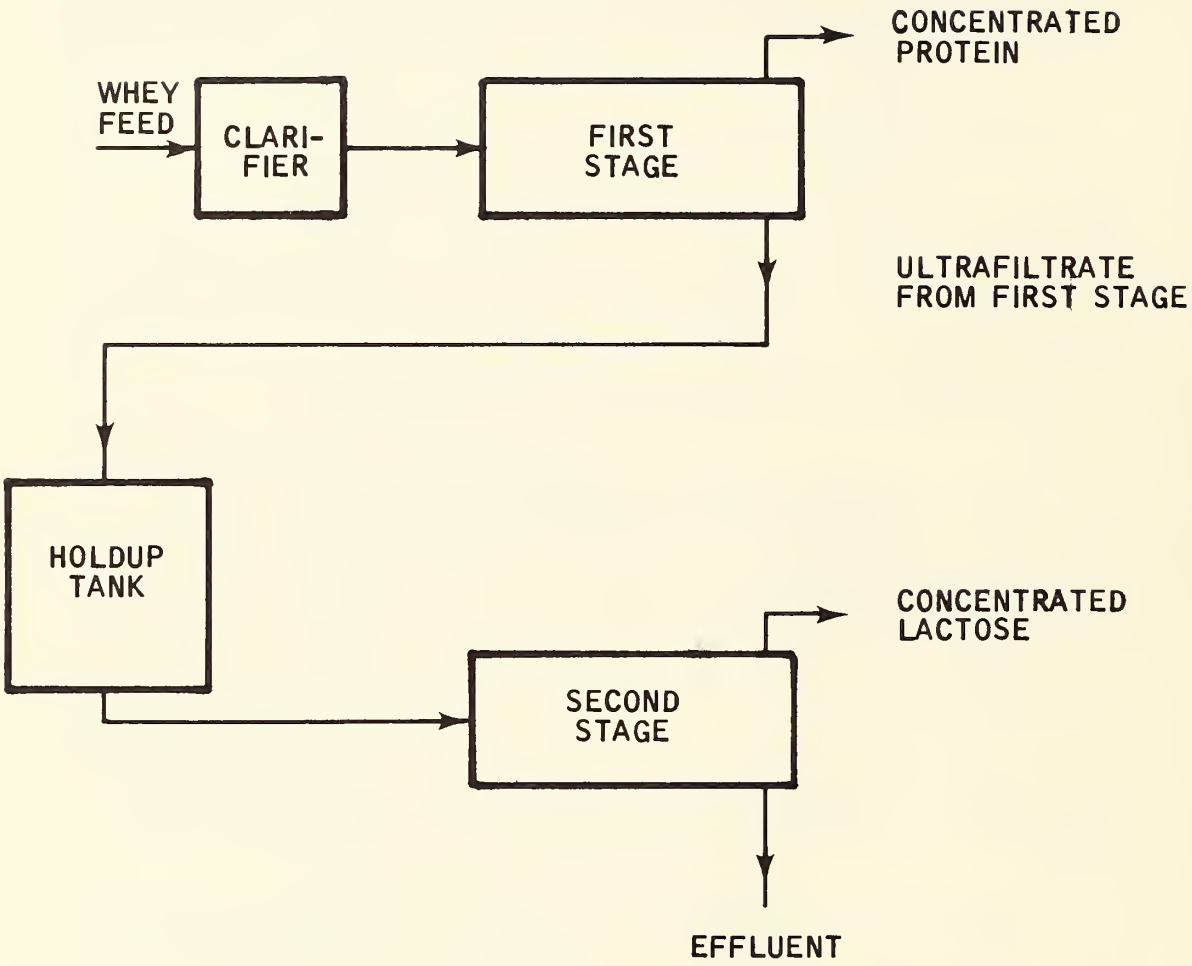

Figure 1.--Flowsheet for two-stage whey ultrafiltration process (2).

TABLE 6.--Composition of fractions from a product containing crystallized lactose[1/]

Sample	Solids, pct.	Lactose		Protein (N x 6.38)		Other	
		Pct.	Pct. of solids	Pct.	Pct. of solids	Pct.	Pct. of solids
Concentrate	53.3	40.4	75.4	10.5	18.7	2.4	5.9
Supernate	27.9	11.0	39.5	13.1	47.0	3.8	13.5
Infranate	29.7	9.5	32.0	16.9	56.8	3.3	11.2
Precipitate	70.0	63.1	90.0	6.5	9.3	.4	.7

1/ Data from Marshall, Dunkley and Lowe (4).

Another promising process for the second stage of fractionation uses a molecular sieve or gel-filtration concept to separate the whey solids (7).

Cost is an important factor that will determine the commercial feasibility of the process. Table 7 summarizes some estimates and the assumptions on which they are based. Another estimate of equipment and operating costs, summarized in figures 2 and 3, compares processing by membrane separation and vacuum evaporation.

TABLE 7.--Cost of concentrating whey by reverse osmosis[1/]

Concentration ratio	Volume (lb./yr.)	Cost	
		Per 1,000 lb. whey	Per lb. H_2O removed
2:1	17,700,000	$ 0.43	$ 0.00092
3:1	9,750,000	.76	.0012
4:1	6,580,000	1.14	.0017

1/ Estimates by McDonough (5) for Havens 3A Membrane 800 p.s.i., 10 hr./day, 300 day/yr., unit cost $19,000 depreciated in 4 years, membranes replaced for $2,500 after 2 years, $300 for energy costs.

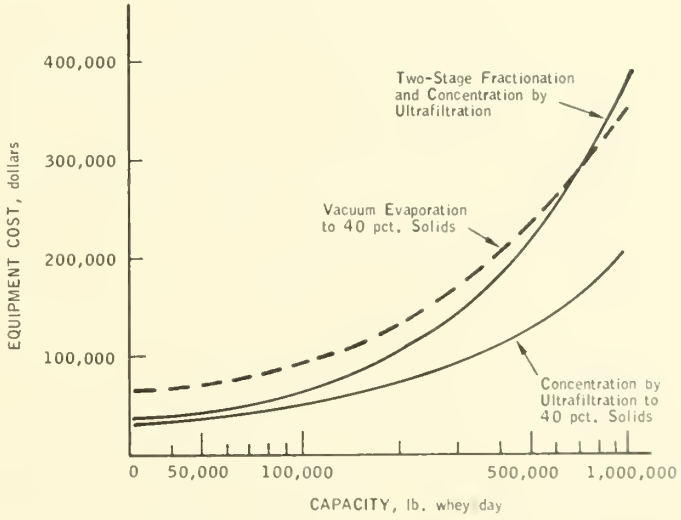

Figure 2.--Equipment costs for concentrating whey byproducts (1).

One of the problems that presently limits application of reverse osmosis to whey processing is a decrease in permeation rate during operation. Figure 4 shows the decrease during 83 hr. of operation of the Wurstack apparatus at 1,400 p.s.i. with Loeb-type membranes. Recent improvements in the flow system in the Wurstack have reduced the drop-off in flux. Also, effectiveness of periodic flushing with water in restoring flux is illustrated in figure 5. In this experiment, the equipment was

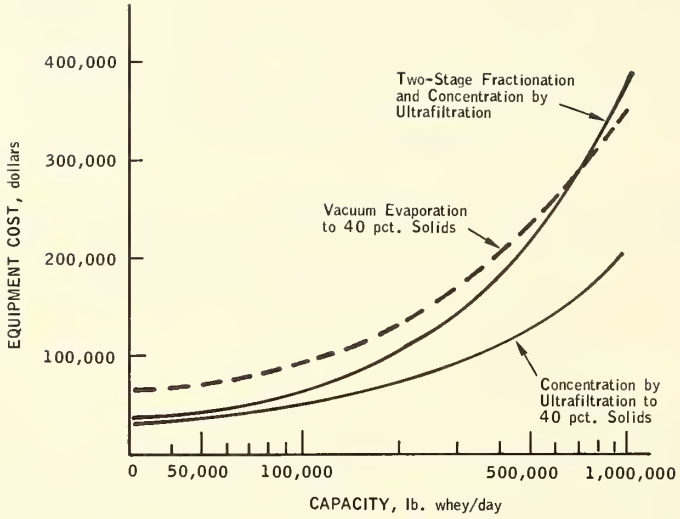

Figure 3.--Operating costs for concentrating whey byproducts (1).

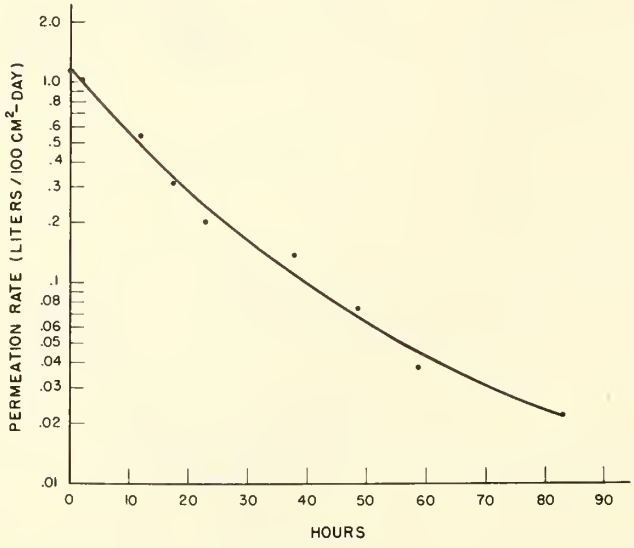

Figure 4.--Semilogarithmic plot of permeation rate against time (5).

operated continuously with whey circulating at about 13-percent total solids except for a 30-min. flush with cold water once a day. The experiment also included tests to determine the influence of operating pressure on flux.

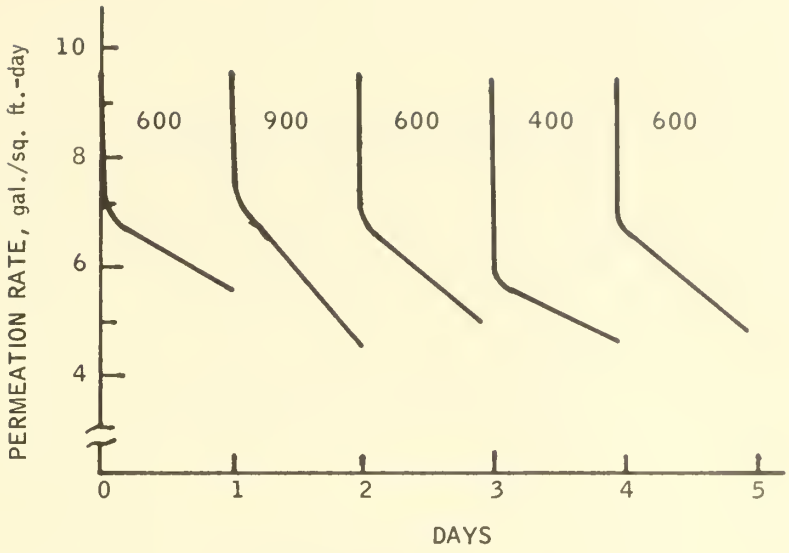

Figure 5.--Influence of flushing with cold water for 30 min. daily on permeation rate (3).

Increasing flow velocity to increase turbulence is recognized as an effective way to minimize concentration polarization and membrane fouling. For example, figure 6 shows that

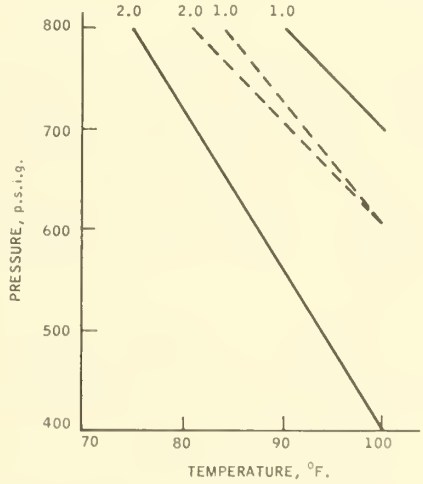

Figure 6.--Minimum operating conditions for flux of 10 gal./sq. ft.-day. Solid lines: 11-19 hr. Broken lines: 28-36 hr. (8).

doubling the flow velocity reduced the operating pressure required to achieve a minimum flux of 10 gal./sq. ft.-day (e.g., at 90°F., from 800 p.s.i. to about 560 p.s.i.). Toward the end of the experiment (28-36 hr., shown by broken lines), changing the velocity had less effect.

The data in figure 7 indicate the relative importance of the whey constituents in contributing the concentration polarization and the decline in flux. When whey was fractionated and

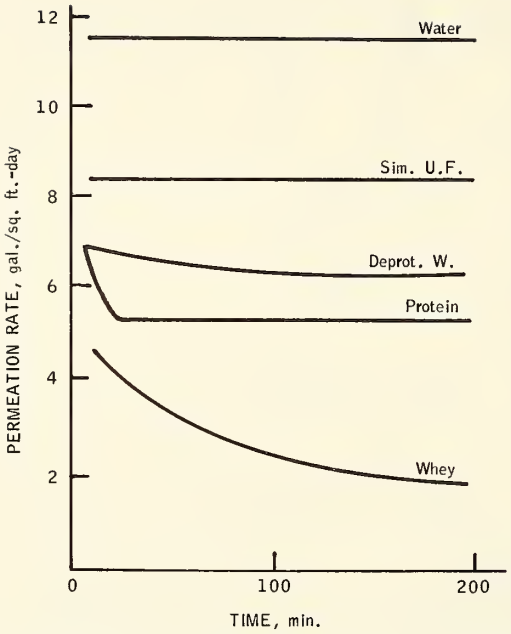

Figure 7.--Influence of composition on permeation rate (6).

permeation rates were determined with individual fractions at concentrations typical of whey, protein caused the greatest decrease in permeation rate. The deproteinized whey, prepared by boiling the whey and clarifying centrifugally to remove the precipitated proteins, still contained large molecules such as proteose-peptone, which probably caused the lower permeation rate than that obtained with the simulated ultrafiltrate, containing milk salts and lactose.

The results in figure 8 emphasize the importance of the whey constituents in determining the permeation rate. Two membranes with marked differences in their water permeation rates gave almost identical permeation rates when used with whey.

The results in figure 9 illustrate the effects of turbulence promoters on permeation rate. The turbulence promoters used in this experiment were rods 5/16 in. outside diameter, with small rings spaced alternately 0.5 and 4.5 in. apart, mounted in the Haven's tubes (0.5 in. inside diameter). For both whey and the solution of whey proteins, the turbulence promoters caused a marked increase in permeation rates.

Zanzig (8) listed the following as key factors affecting the industrial potential of the process:

1. The ultimate membrane life and the flux performance stability over this life.

2. Membrane fouling in a cheese whey system.
3. Sanitation and sanitary design.
4. Membrane replacement costs.
5. Practical equipment from an operating point-of-view.

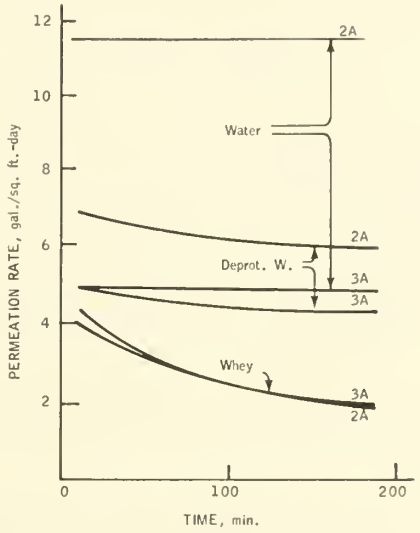

Figure 8.--Influence of membrane on permeation rate (6).

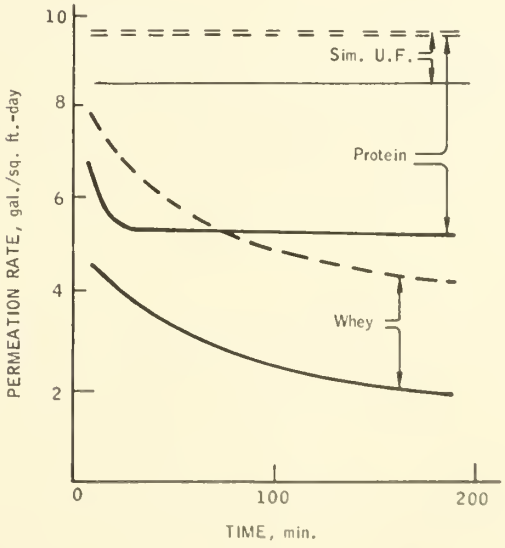

Figure 9.--Influence of turbulence promoters on permeation rate (solid lines--without turbulence promoters; broken lines--with turbulence promoters) (6).

In view of the intensive research and development activity on application of reverse osmosis and ultrafiltration to whey processing, more reliable information regarding these factors should become available in the near future. Many who are knowledgeable in this field consider that reverse osmosis holds promise for the commercial concentration and fractionation of whey.

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TREATMENT OF WASTE STREAMS FROM FOOD PROCESSING

Walter A. Mercer, Associate Director
National Canners Association Laboratories
Berkeley, Calif.

[Presented by Jack W. Ralls
NCA Western Research Laboratory
Berkeley, Calif.]

We at the Western Research Laboratory of the National Canners Association are pleased to act as cosponsors of this conference on "Reverse Osmosis in Food Processing." As you all recognize, the technology applied to the preservation of foods is changing continuously and rapidly. It appears certain to us that reverse osmosis will have important applications in food processing and in liquid waste management practices.

Equipment research and development is not a major responsibility in the research and service work which we perform for the members of the National Canners Association. However, we must follow processing equipment developments closely, and we do evaluate new processing equipment in order to provide useful service to our members.

Future uses of reverse osmosis.--The most probable initial application of reverse osmosis for food processing will be in the production of juice concentrates. We feel that the use of reverse osmosis is promising for the preparation of tomato puree, fruit nectars, and fruit and vegetable juice concentrates. We see several advantages in concentration of juices by reverse osmosis. The most important is the higher degree of retention of flavor components in a reverse osmosis operation as compared to heated, vacuum pan concentrators.

The very serious problems facing the food processing industry in disposing of byproduct wastes has made us acutely conscious of the need for the total systems approach in considering any change in food production operations. The impact of any new technique of food processing must be evaluated in terms of multiple parameters including cost of production, rate of production, sanitation, product quality, and waste disposal of byproducts. The concentration of juices by reverse osmosis will produce a byproduct water of good quality which could be used for raw product washing or plant cleanup before discharge. The problem of disposal or reconditioning of used membranes from reverse osmosis units is one deserving attention.

There is considerable current interest in pre-processing of fruits and vegetables in the field. This trend is under consideration because of the expanding use of machine harvesting and of the increasingly more difficult waste disposal of byproducts from crops when conducted in urban environments. Reverse osmosis units may have important advantages in field pre-processing operations because of their low steam requirements and the option for ready transport to various field locations.

Another likely feature of juice concentration conducted in a plant by reverse osmosis equipment is in a continuous operation where the product is sterilized and packaged by an aseptic canning or barreling unit.

There will be a reduction in the volume of water needed for condenser cooling when a reverse osmosis unit replaces a vacuum pan concentrator.

The water from evaporator condenser cooling is reused in many plants for raw product washing. However, in some plants the condenser water creates a problem due to possible thermal pollution of receiving waters or hydraulic overloading of sewage treatment systems.

Treatment of waste streams by reverse osmosis.--Even after many years of research by NCA and many other laboratories, there are still food processing waste disposal problems which have no economical solution. There are a number of waste disposal situations where reverse osmosis may provide these urgently needed solutions.

As Professor Dunkley has emphasized, it is desirable to recover valuable materials by treatment of waste streams to recover at least part of the cost of the operation.

A very serious waste disposal problem is facing the producers of olives, cherries, pickles, and sauerkraut. Large volumes of saline wastes are generated by the production of these products. Special handling of these saline wastes are required because of the non-biodegradable nature of sodium chloride and the low limits for salts in receiving water soon to be enforced by regulatory agencies.

Food processing plants located near salt water bays have a disposal advantage not enjoyed by inland plants. These latter plants are currently disposing of saline wastes by slow discharge into rivers or municipal treatment plants where dilution will reduce the salt concentration to acceptable levels, injection of salt solutions into sealed underground reservoirs, or discharge

to solar evaporation ponds. None of these disposal methods are completely satisfactory because of cost considerations or constantly reduced assimilation capacity of receiving waters.

One potential solution to the discharge of strong brines used to store and ferment olives, cucumbers, and cabbage is the reconditioning and reuse of these brines. The NCA has a current project, based on results obtained here at the Western Utilization Research and Development Division, which is evaluating the use of activated carbon for reconditioning olive storage brines containing 4-10 percent sodium chloride with BOD's as high as 15,000 p.p.m. The reuse of reconditioned brine on a commercial scale looks very promising from a feasibility standpoint, but no representative cost figures have been derived as yet.

Our most recent direct involvement in applications of reverse osmosis methodology was in the analysis of samples of membrane filtered Spanish olive brines. These results showed that the color, chemical oxygen demand, and total bacterial count were substantially reduced after membrane filtration while salt content, pH and total acidity were only slightly changed. The flux produced by the membrane filtration unit had good potential for reuse as storage and fermentation brine. The concentrate had a high BOD and low salt content which made it an acceptable discharge to a municipal sewage treatment plant. The results of this exploratory study were reported by C. S. Britton of Havens Industries at the Technical Conference of the California Olive Association, June 24-26, 1968.

A difficult waste for the olive industry to manage is the large volume of processing waters from the lye curing, washing, and aeration operations. At one plant the volume of this waste stream is 400,000 gal. per day. A typical composition of this composite waste is: suspended solids, 68 p.p.m.; COD, 6,400 mg./liter; pH, 10.6; and salt content 0.2 percent (or 2,000 p.p.m.). This waste stream would be amenable to treatment by reverse osmosis. A purified water could be produced which could be recycled. The saline concentrate which did not pass the membrane could possibly be reconditioned by activated carbon treatment and used as storage brine.

Seafoods are another commodity area where a potentially valuable byproduct could be produced by reverse osmosis. The production of fishmeal and fish oil from menhaden, herring, tuna, and other fish has as a byproduct a press-liquor known as stickwater. This liquid waste contains soluble fish components and is particularly rich in peptides, amino acids, and vitamins. The stickwater is usually concentrated to a 50-percent-solids content material in continuous, vertical, triple-effect vacuum evaporators.

The condensed fish solids has a market as an animal feed. The concentration of stickwater is not without problems, especially in small operations where batch-evaporation in steam-jacketed kettles is used. The milder conditions used in concentration by reverse osmosis should yield a more nutritious fish soluble concentrate with higher vitamin content.

The canning and freezing of fruits generates large volumes of liquid waste having dissolved sugars and organic acids. A concentrate of fruit processing water has promise as a substrate for fermentations producing valuable materials such as glutamic acid. Many municipalities are charging "sewer taxes" to dischargers of liquid wastes having high BOD content. Sums equivalent to "sewer taxes" on liquid wastes could be applied to partly underwrite product recovery systems which may appear uneconomic on initial consideration.

We would encourage manufacturers and suppliers of reverse osmosis equipment to investigate the application of this technique to every type of food processing waste. There is every prospect that a number of economically viable solutions to problems of food processing waste disposal would result from such investigations.

It has been a pleasure to participate in this conference. If the National Canners Association can be of assistance to you, as for example in supplying information on composition of waste streams from food processing, please call on us at any time.

REPORT OF INFORMAL DISCUSSIONS

L. Frank Ginnette, Chemical Engineer
Western Utilization Research and Development Division
Agricultural Research Service, USDA, Albany, Calif.

The afternoon session was given over to an informal discussion of various aspects of reverse osmosis. Questions and comments were solicited from the conference participants, as well as from the speakers of the morning. The discussion was too lengthy to be reproduced here in its entirety. A brief summary is given below.

Membranes.--The speakers of the morning session had referred in the main to the use of anisotropic cellulose acetate membranes. In the afternoon, several equipment suppliers

mentioned other types, among which were isotropic cellulose acetate, nylon, and glass. These membranes vary in the degree to which they reject various solutes and also in the rate at which they pass water under a given hydrostatic pressure. In general, it can be stated that there is an inverse relation between solute rejection and water permeability, but the relation is extremely ill-defined. The material of which the membrane is composed, its dimensions, whether or not it is isotropic, and many other factors influence the selectivity and permeability.

Some suppliers have chosen to manufacture membranes but not pressure vessels and the ancillary hardware. Flat sheets of membrane of limited width, but of essentially unlimited length, are available from these suppliers. Others have elected to supply not only the membrane but the support structures as well, and in these instances a wider variety of physical forms is available. Among these are "large bore" tubular membranes which are to be pressurized from the inside while restrained in a "straight-jacket," and "small bore" tubes which are pressurized from the outside.

Some suppliers have preferred to use relatively high-flux membranes in order to make do with the least amount of membrane surface; others have preferred to use low-flux membranes but have contrived to incorporate large surface area into a relatively small volume.

Membrane life.--The service life of membranes is not very well known because most applications of reverse osmosis are of rather recent origin. One supplier gave an example in which anisotropic cellulose acetate membranes were going into their fourth year of service on a feed stream consisting of calcium sulfite liquor (pH 3.5). One or two other examples of a membrane having survived several months of service were given. While relatively little is known on this subject at the present time, a note of cautious optimism appears to be in order.

Sanitation.--Several bactericidal agents have been found that can be used to sterilize reverse osmosis equipment without damaging the membranes. The only one mentioned by name was chlorine, which, it was said, can be used at concentrations up to 10 p.p.m. A supplier of glass membranes pointed out that these can stand sterilizing temperatures.

Concentration polarization.--One of the speakers reported an instance in which membranes having different permeabilities, as measured by their ability to pass pure water at a given pressure, gave identical fluxes when used on another material (in this case, whey).

This was conjectured to be due to the phenomenon of concentration polarization, which is the accumulation of a static layer of highly concentrated material near the membrane surface. The phenomenon results from the separation that occurs at the membrane where water passes on through, leaving the solutes behind. These solutes must be carried into the main stream of fluid by diffusion or convection; otherwise they interfere with the delivery of water to the membrane. In some instances the rate of permeation is believed to be controlled entirely by the efficiency with which this interfering layer can be washed away. The situation may be aggravated by increases in fluid viscosity that may accompany the increase in solids content.

Hardware.--A number of pieces of reverse-osmosis hardware were described and some were demonstrated. These included plate-and-frame type devices, using flat membranes; a "jelly roll" type, in which an assembly consisting of membrane, flow channels, and spacers is literally rolled up into a compact bundle; an all-plastic device in which liquid flows under pressure inside 1/2-in. diameter tubular membranes, and fiber-bundle devices in which the pressurized fluid flows over the exterior of a bundle of tiny hollow fibers of isotropic membrane material.

Each of these devices represents a somewhat different approach to the problem of efficient reverse-osmosis hardware, and each has its peculiar advantages and disadvantages. Some are designed only for low-pressure work, and thus might be suited to treatment of effluent or other dilute streams. Some, notably the all-plastic and fiber-bundle type, are suited to intermediate-pressure work (up to 600 p.s.i.), such as is needed in concentrating egg white or whey.

Others can withstand upwards of 1,000 p.s.i. and thus might be suitable for high osmotic-pressure applications, such as concentration of fruit juices.

A potential user of the reverse osmosis process has a wide selection of equipment to draw from.

ATTENDANCE

J. A. Abbott
FMC Corp.
1185 Coleman Ave.
Santa Clara, Calif. 95052

Lyle Allen
Foamat Foods Corp.
P. O. Box 725
Corvallis, Oreg. 97330

J. L. Alm
Safeway Stores, Inc.
2538 Telegraph Ave.
Oakland, Calif. 94612

Kenneth E. Anderson
Havens International
8133 Aero Drive
San Diego, Calif. 92123

Jerry M. Attebury
Swanson & Associates, Inc.
1710 N. Douglas Drive
Minneapolis, Minn. 55422

Robert C. Ayers
Chas. Pfizer & Co., Inc.
Eastern Point Road
Groton, Conn. 06340

Guy N. Baldwin, Jr.
Paul Masson Vineyards
P. O. Box 96
Saratoga, Calif. 95070

Robert Battey
FMC Corp.
P. O. Box 580
Santa Clara, Calif. 95052

R. N. Berry
Continental Can Co., Inc.
685 "A" St.
Hayward, Calif. 94541

Thomas D. Birchall
G. W. Hume Co.
P. O. Drawer 710
Turlock, Calif. 95380

J. M. Bonnell
Tropicana Products, Inc.
P. O. Box 338
Bradenton, Fla. 33505

Charles M. Buchzik
FMC Corp.
1185 Coleman Ave.
Santa Clara, Calif. 95052

Charles D. Buss
Tillie Lewis Foods, Inc.
Drawer J
Stockton, Calif. 95201

Bernard J. Butler
210 California St.
San Francisco, Calif. 94111

P. A. Cantor
Aerojet-General Corp.
9200 East Flair Drive
El Monte, Calif. 91734

Lee Chambers
Carnation Research Laboratories
8015 Van Nuys Blvd.
Van Nuys, Calif. 91412

Raymond M. Chappel
American Standard
P. O. Box 2003
New Brunswick, N. J. 08903

William J. Chvala
Coca-Cola
P. O. Drawer 1734
Atlanta, Ga. 30301

T. J. Connelly
Del Monte Corp.
2600 - 7th St.
Berkeley, Calif. 94710

Marshall Cook
Battelle - Northwest
Box 15
Richland, Wash. 99352

John Cotter
E. I. du Pont de Nemours & Co.
P. O. Box 525
Wilmington, Del. 19899

Vijay N. Das
Chemical Engineering Department
University of California
Davis, Calif. 95616

James C. Davis
Dow Chemical Research Laboratory
2800 Mitchell Drive
Walnut Creek, Calif. 94598

F. De Maria
American Machine & Foundry Co.
689 Hope St.
Stamford, Conn. 06906

George DeMedeiros
Dairyman's Cooperative
Creamery Assoc.
400 South "M" St.
Tulare, Calif. 93274

John S. DeMurigy
American Standard
P. O. Box 2003
New Brunswick, N. J. 08903

Max R. Dietz
Gerber Products Co.
445 State St.
Fremont, Mich. 49412

James A. Downer
Canada Department of Industry
Place De Ville
Ottawa, Ontario, Canada

Donald Hoyt Doud
E. I. du Pont de Nemours & Co.
Jackson Laboratory
P. O. Box 525
Wilmington, Del. 19899

D. R. Downing
Libby McNeill & Libby
200 S. Michigan Ave.
Chicago, Ill. 60604

Richard Drushella
Sta-Rite Ind., Inc.
Delavan, Wis. 53115

W. L. Dunkley
University of California
209 Roadhouse Hall
Davis, Calif. 95616

H. N. Dunning
General Mills, Inc.
James Ford Bell Technical Center
9000 Plymouth Ave.
Minneapolis, Minn. 55427

R. G. Estabrooks
Carnation Research Laboratories
8015 Van Nuys Blvd.
Van Nuys, Calif. 91412

Gary J. Fallick
Amicon Corp.
25 Hartwell Ave.
Lexington, Mass. 02173

Robert C. Farris
Borden, Inc.
1325 Potrero Ave.
San Francisco, Calif. 94119

Robert A. Fiedler
Dorr-Oliver, Inc.
77 Havemeyer Lane
Stamford, Conn. 06904

Eric Forbes
Dorr-Oliver, Inc.
77 Havemeyer Lane
Stamford, Conn. 06904

L. H. Francis
Foremost Foods Co.
111 Pine St.
San Francisco, Calif. 94111

Paul U. Frei
United Vintners, Inc.
P. O. Box 1
Asti, Calif. 95413

E. H. Fults
Fults Chemical, Inc.
P. O. Box 239
Tulare, Calif. 93274

James H. Garrison
Mid-America Dairymen, Inc.
P. O. Box 1837 S.S.S.
Springfield, Mo. 65805

Altanir J. Gava
University of California
A-8 Orchard Park
Davis, Calif. 95616

Blair C. Gibbons
Electro-Membrane Systems, Inc.
2532 Pulgas Ave.
Palo Alto, Calif. 94303

L. C. Gilde
Campbell Soup Co.
375 Memorial Ave.
Camden, N. J. 08101

Daniel C. Gillespie
Dorr-Oliver, Inc.
77 Havemeyer Lane
Stamford, Conn. 06904

C. Gur-Arich
Del Monte Corp.
215 Fremont St.
San Francisco, Calif. 94105

G. A. Guter
McDonnell-Douglas
Astropower Laboratory
2121 Campus Drive
Newport Beach, Calif. 92660

William G. Halversen
California Canners and Growers
312 Stockton Ave.
San Jose, Calif. 95124

Frank Hammond
Sebastopol Co-op
P. O. Box 241
Sebastopol, Calif. 95472

Delbert K. Harmann
Mid-America Dairymen, Inc.
P. O. Box 1837 S.S.S.
Springfield, Mo. 65805

John Harper
University of California
Agricultural Engineering Dept.
Davis, Calif. 95616

Paul D. Hass
Swanson & Associates, Inc.
1710 N. Douglas Drive
Minneapolis, Minn. 55422

Ed Hirschberg
E. Hirschberg Freeze Drying
179 Starlite St.
South San Francisco, Calif. 94080

Sus Ikuta
Bio-Rad Labs
32nd and Griffin Avenues
Richmond, Calif. 94804

O. L. Ingebretsen
Del Monte Corp.
2600 - 7th St.
Berkeley, Calif. 94710

R. M. Jorgensen
Del Monte Corp.
2600 - 7th St.
Berkeley, Calif. 94710

T. Kasandjieff
Dairyman's Cooperative
Creamery Assoc.
400 South "M" St.
Tulare, Calif. 93274

Allen Katsuyama
National Canners Assoc.
1950 - 6th St.
Berkeley, Calif. 94710

Chester E. Kean
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Crockett, Calif. 94525

D. M. Kelly
De Laval Separator Co.
1815 Rollins Road
Burlingame, Calif. 94010

C. Judson King
Department of Chemical Eng.
University of California
Berkeley, Calif. 94720

Fred A. Krantz
General Mills, Inc.
James Ford Bell Technical Center
9000 Plymouth Ave.
Plymouth, Minn. 55427

Seymour S. Kremen
Gulf General Atomic, Inc.
P. O. Box 608
San Diego, Calif. 92112

O. J. Krett
National Dairy Products Corp.
801 Waukegan Road
Glenview, Ill. 60025

D. H. Larson
Sta-Rite Ind., Inc.
Box 622
Delavan, Wis. 53115

Fred B. Leonard
Havens International
8133 Aero Drive
San Diego, Calif. 92123

N. E. Liles
Tri-Valley Growers
100 California St.
San Francisco, Calif. 94111

Toh-Hoy Lim
University of California
1224 "F" St., #16
Davis, Calif. 95616

Ray Long
Dow Chemical Co.
Walnut Creek, Calif. 94596

Harold K. Lonsdale
Gulf General Atomic, Inc.
P. O. Box 608
San Diego, Calif. 92112

Edmar Salvador Loureiro
University of California
Food Science and Technology Dept.
Davis, Calif. 95616

Robert D. Lukacik
Ionics, Inc.
65 Grove St.
Watertown, Mass. 02172

Salvatore Maggio
M. Maggio Co.
1100 S. 11th St.
Philadelphia, Pa. 19147

H. I. Mahon
Dow Chemical Co.
2800 Mitchell Drive
Walnut Creek, Calif. 94598

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Eastman Chemical Products, Inc.
Kingsport, Tenn. 37662

Serop Manjikian
Universal Water Corp.
1638 Coast Blvd.
Del Mar, Calif. 92014

Paul G. Marshall
Vacu Dry Co.
1311 - 63rd St.
Emeryville, Calif. 94608

Bromley M. Mayer
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231 E. 23rd St.
Los Angeles, Calif. 90011

H. D. Mead
DeLaval Separator Co.
1815 Rollins Road
Burlingame, Calif. 94010

Reginald E. Meade
The Pillsbury Co.
Research Laboratory
311 Second St., S.E.
Minneapolis, Minn. 55414

R. E. Meece
Carnation Research Laboratories
8015 Van Nuys Blvd.
Van Nuys, Calif. 91412

Charles P. Minning
University of California
Sea Water Conversion Laboratory
1301 S. 46th St.
Richmond, Calif. 94804

Lee Nutting
Hills Brothers Coffee, Inc.
2 Harrison St.
San Francisco, Calif. 94119

D. E. O'Connor
Procter & Gamble
6000 Center Hill Road
Cincinnati, Ohio 45224

Harold K. Olson
Land O'Lakes Creameries, Inc.
1243 N. Eleventh St.
Eau Claire, Wis. 54701

Dermot A. O'Sullivan
Chemical & Eng. News
57 Post, Suite 703
San Francisco, Calif. 94104

Warren G. Palmer
FMC Corp.
P. O. Box 580
Santa Clara, Calif. 95052

E. J. Parsi
Ionics, Inc.
65 Grove St.
Watertown, Mass. 02172

H. J. Pederson
Foremost Foods Co.
6363 Clark Ave.
Dublin, Calif. 94566

D. F. Percival
Crown Zellerback Corp.
2101 Williams St.
San Leandro, Calif. 94577

Wendell Perry
American Standard
40 W. 40th St.
New York City, N. Y. 10018

Roy W. Porter
Anheuser-Busch, Inc.
721 Pestalozzi
St. Louis, Mo. 63118

Ronald E. Pyle
Miles Laboratories, Inc.
900 - 19th St.
Granite City, Ill. 62040

Jack Quinton
National Can Corp.
290 Division St.
San Francisco, Calif. 94103

Jack W. Ralls
National Canners Assoc.
1950 - Sixth St.
Berkeley, Calif. 94710

Robert D. Rands
Mallinckrodt Chemical Works
P. O. Box 5439
St. Louis, Mo. 63160

Sutton Redfern
Standard Brands Inc.
The Fleischmann Laboratories
Betts Ave.
Stamford, Conn. 06902

Harold Redsun
Del Monte Corp.
2600 - 7th St.
Berkeley, Calif. 94710

James D. Richart
R. J. Reynolds Tobacco Co.
Winston Salem, N. C. 27101

Duane Rohrer
Tri-Valley Growers
P. O. Box 948
Modesto, Calif. 95352

Walter W. Rose
National Canners Assoc.
1950 Sixth St.
Berkeley, Calif. 94710

M. E. (Gene) Rowley
Eastman Kodak Co.
Kodak Park
Rochester, N. Y. 14603

M. W. Saba
American Standard
P. O. Box 2003
New Brunswick, N. J. 08903

Stan Salsbury
Dairyman's Cooperative
Creamery Assoc.
400 South "M" St.
Tulare, Calif. 93274

Francis R. Saunders
Lamb-Weston, Inc.
12977 S.W. 66th St.
Portland, Oreg. 97223

Burton A. Schaffer
General Foods Corp.
250 North St.
White Plains, N. Y. 10602

Mrs. R. Schwab
University of California
Sea Water Conversion Laboratory
1301 South 46th St.
Richmond, Calif. 94804

F. R. Senti
ARS, USDA
Washington, D.C. 20250

Henry Shanfield
Polymetrics
810 Cherry Lane
San Carlos, Calif. 94070

James Siciliano
EURDD, ARS, USDA
600 East Mermaid Lane
Philadelphia, Pa. 19188

W. Eugene Skiens
The Dow Chemical Co.
2800 Mitchell Drive
Walnut Creek, Calif. 94598

Gerald L. Sloan
Lamb-Weston, Inc.
12977 S.W. 66th St.
Portland, Oreg. 97223

K. S. Spiegler
Sea Water Conversion Laboratory
1301 South 46th St.
Richmond, Calif. 94804

Matthew M. Stawiarski
Central Soya Company, Inc.
1825 N. Laramie Ave.
Chicago, Ill. 60639

R. W. Stephenson
General Mills, Inc.
9000 Plymouth Ave.
Minneapolis, Minn. 55427

Thomas A. Swinford
Knudsen Corp.
231 E. 23rd St.
Los Angeles, Calif. 90011

Robert S. Timmins
Abcor, Inc.
341 Vassar St.
Cambridge, Mass. 02139

E. G. Tonn
Crown Zellerback Corp.
2101 Williams St.
San Leandro, Calif. 94577

Bernard A. Twigg
Department of Horticulture
University of Maryland
College Park, Md. 20742

Richard T. Tyndall
Dole Co.
P. O. Box 3380
Honolulu, Hawaii 96801

J. Clyde Underwood
EURDD, ARS, USDA
600 Mermaid Lane
Philadelphia, Pa. 19118

J. E. Ward
Safeway Stores, Inc.
2538 Telegraph Ave.
Oakland, Calif. 94612

Neal S. Weeks
Safeway Stores, Inc.
612 W. 5th St.
Hanford, Calif. 93230

N. A. Weil
FMC Corp.
Box 760
San Jose, Calif. 95106

Ron Weiss
Miles Laboratories, Inc.
900 - 19th St.
Granite City, Ill. 62040

Charles Wendland
CAW Engineering
2532 Pulgas Ave.
Palo Alto, Calif. 94303

L. G. Williams
Del Monte Corp.
215 Fremont St.
San Francisco, Calif. 94116

C. W. Wilson
1050 W. Francis St.
Ontario, Calif. 91762

Robert L. Winslow
Safeway Stores, Inc.
2538 Telegraph Ave.
Oakland, Calif. 94612

C. D. Wintermantel
Del Monte Corp.
2600 - 7th St.
Berkeley, Calif. 94710

C. C. Witmer
Tri-Valley Growers
P. O. Box 948
Modesto, Calif. 95352

Burt Zabin
Biorad Labs
32nd and Griffin
Richmond, Calif. 94804

Robert R. Zall
Crowley's Milk Co., Inc.
145 Conklin Ave.
Binghamton, N. Y. 13902

Charles E. Zanzig
Foremost Foods
Box 2287
Dublin, Calif. 94566

Western Utilization Research and Development Division
Agricultural Research Service, USDA

J. Peter Clark
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UNITED STATES DEPARTMENT OF AGRICULTURE
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